Application Note WDXRF1088

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XRF1088 - Boron Analysis In gass powder

Introduction

Boron oxide (or boric acid) is an important compound for making glass. It is added to glass to decrease the softening temperature without changing the thermal expansion coefficient or chemical durability for glass. The boron element line, B-Kα, has a very long wavelength, making boron analysis by X-ray fluorescence (XRF) spectrometry difficult. Using a high power X-ray tube and an excellent synthetic multilayer analyzer has achieved excellent analysis results of boron oxide (B_2O_3) in glass by XRF.

This Application Note demonstrates quantitative analysis results of B_2O_3 in glass by XRF.

Instrument

The ZSX Primus IV, a tube-above sequential wavelength dispersive X-ray fluorescence (WDXRF) spectrometer, is equipped with a 4 kW X-ray tube with a Rh target featuring an ultra-thin beryllium window, and four analyzing crystals covering ^{8}O to ^{96}Cm. In this analysis, the synthetic multilayer analyzer RX85 (optional) is also mounted in the spectrometer.

The ZSX Primus IV is optimized for routine analysis of powder samples. The system's tube-above optics, variable vacuum speed, powder trap and pre-evacuation chamber enable secure analysis of powder samples, and reduce maintenance by preventing pressed powder pellets from breaking apart and falling into the optics, and by protecting the vacuum pumps and magnetic valves from fine particles scattered from samples.

The system software is designed for ease of use in routine analyses. The “Flowbar” in quantitative analysis guides users through calibration. The “Sample ID Table” and the “Program Operation” help operators carry out daily analysis.

Measurement condition

Measurements were performed on the ZSX Primus IV with a 4 kW X-ray tube operating at 30 kV and 120 mA using the RX85 synthetic multilayer and the S8 high-sensitivity Soller slit (optional). The counting time was 100 seconds each for peak and background.

The RX85, a new multilayer analyzer, has 1.3 times as high sensitivity as the previous multilayer, RX75.

For boron analysis, APC (Auto Pressure Control) was used. This function keeps the vacuum levels in the sample and optical chambers constant during measurement. Without APC, changes in the vacuum level in the X-ray path would cause the X-ray intensities of B-Kα to fluctuate because of the characteristic line's long wavelength.

Pb-Lβ1 was also measured with LiF(200) and the S2 high-resolution Soller slit for overlap correction.

Standard and sample preparation

NIST SRM92, SRM1411 and SRM1412, certified reference materials of glass, were used for calibration, together with high-purity (99.99%) silica as a blank. SRM1411 and SRM1412 were ground with a vibration mill using a tungsten-carbide container. The powders of the NIST reference materials and the high-purity silica, pre-dried at 110 °C, were pressed into PVC rings (I.D. 32 mm) at 150 kN.

Analysis result

Figure 1 shows the calibration curve obtained with the NIST reference materials of glass and the high-purity silica. Overlap correction by Pb was applied. Calibration accuracy was 0.063 mass% and the estimated lower limit of detection (LLD) is 0.056 mass% for boron oxide (0.017 mass% for boron). Calibration accuracy is calculated using the following formula:

$$Accuracy = \sqrt{\frac{\sum_i (C_i - \hat{C}_i)^2}{n-2}}$$

C_i: calculated value of standard sample
$\hat{C}_i$: reference value of standard sample
n : number of standard samples.

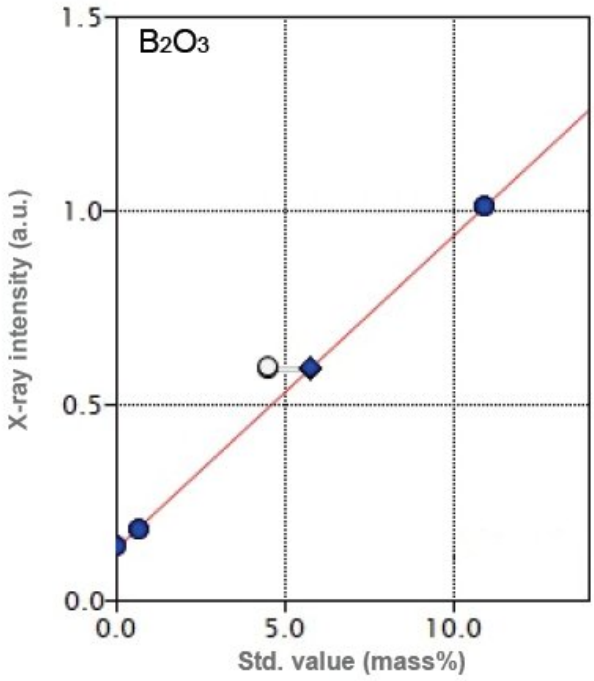

Figure 1: Boron oxide calibration curve

Table 1: Repeatability test results for B_2O_3 (mass%) .

Sample	NIST SRM92	NIST SRM1411
n=1	0.78	10.88

2	0.74	10.98
3	0.73	10.98
4	0.76	10.95
5	0.77	10.90
6	0.76	11.02
7	0.73	10.94
8	0.74	10.94
9	0.78	10.91
10	0.79	10.94
Average	0.76	10.94
Standard deviation	0.021	0.041
RSD	2.78%	0.38%

The LLD is calculated as three times the theoretical standard deviation of the blank.

Repeatability tests (10 runs), during which the samples stayed inside the sample chamber, were carried out by running NIST SRM92 (B_2O_3 0.70 mass%) and SRM1411 (B_2O_3 10.94 mass%) as analysis samples. The test results are shown in Table 1.

Effects of APC function in boron analysis

Auto Pressure Control (APC) is a function that stabilizes the degree of vacuum in the spectrometer's chambers during measurement. During all the analytical measurements in this Application Note, the APC was kept on.

Figure 2 plots the results of repeatability tests by 10 runs with APC on and off. With APC off, the degree of vacuum in the sample and optical chambers is reducing, which increases the X-ray intensity of B-Kα. This element line has such a long wavelength that the X-ray intensities fluctuate with even small changes in vacuum level.

The APC can achieve high precision for element lines with very long wavelength, such as C-Kα, B-Kα, or Be-Kα.

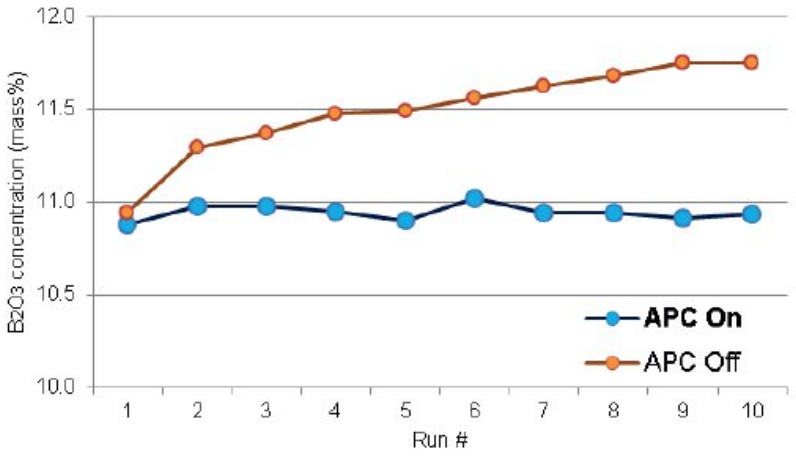

Figure 2: Comparison in stability with APC on and off

Conclusion

Boron oxide in glass can be routinely analyzed on the ZSX Primus IV with a 4 kW X-ray tube with a Rh target featuring an ultra-thin beryllium window, and the synthetic multilayer RX85 by pressing glass power samples into pressed powder briquettes.

The APC function of the ZSX Primus IV keeps the vacuum levels in the sample and optical chambers constant. This function works very well for the measurement of element lines with very long wavelengths, such as C-Kα, B-Kα, or Be-Kα. In this Application Note, high precision in boron oxide analysis in glass powder is reported.

Related products

ZSX Primus IV

High power, tube above, sequential WDXRF spectrometer wi th new ZSX Guidance expert system software